

[\[Return to Article\]](#)**Table 1.** Multidimensional NMR experiments

Experiment (acquisition of central peaks)*	Indirect [†] dimension(s)	<i>t</i> _{max} , ms; complex points	Measurement time, h	Relative sensitivity (peak pairs/ central peaks)
Sequential backbone connectivities (3D spectra)				
<u>H</u> ^{α/β} <u>C</u> ^{α/β} (CO)NHN	$\omega_1(^{13}\text{C}^{\alpha/\beta}/^1\text{H}^{\alpha/\beta})$	6.3; 95	9.2	(0.56/0.34)
(¹³ C)	$\omega_2(^{15}\text{N})$	21.5; 28		
<u>HACA</u> (CO)NHN	$\omega_1(^{13}\text{C}^{\alpha}/^1\text{H}^{\alpha})$	6.5; 54	5.4	(1.00 [‡] /0.81)
(¹³ C)	$\omega_2(^{15}\text{N})$	21.5; 28		
Intraresidual backbone connectivities (3D spectra)				
<u>HNNCAHA</u>	$\omega_1(^{13}\text{C}^{\alpha}/^1\text{H}^{\alpha})$	6.6; 51	5.0	(0.41/0.27)
(INEPT)	$\omega_2(^{15}\text{N})$	21.5; 28		
<u>H</u> ^{α/β} <u>C</u> ^{α/β} COHA	$\omega_1(^{13}\text{C}^{\alpha/\beta}/^1\text{H}^{\alpha/\beta})$	6.3; 95	10.0	(0.22/0.11)
(¹³ C)	$\omega_2(^{13}\text{C}=\text{O})$	17.8; 32		
HNNCACB	$\omega_1(^{13}\text{C}^{\alpha/\beta})$	6.6; 56	8.0	(0.56)
	$\omega_2(^{15}\text{N})$	21.5; 28		
Intra- and sequential-backbone connectivities (3D spectrum)				
HNN <u>(CO,CA)</u>	$\omega_1(^{13}\text{C}^{\alpha}/^{13}\text{C}=\text{O})$	8.0/16.0 [§] ; 54	5.5	(0.54/1.41)
(INEPT)	$\omega_2(^{15}\text{N})$	21.5; 28		
Assignment of aliphatic resonances (3D spectra)				
<u>HCCH-COSY</u>	$\omega_1(^{13}\text{C}/^1\text{H})$	6.3; 95	6.2	(0.34/0.25)
(¹³ C)	$\omega_2(^{13}\text{C})$	6.4; 20		
<u>HCCH-TOCSY</u> [¶]	$\omega_1(^{13}\text{C}/^1\text{H})$	6.3; 95	7.0	(0.19/n.d.)
(¹³ C)	$\omega_2(^{13}\text{C})$	6.4; 20		

Assignment of aromatic resonances (2D spectra)				
<u>HBCB(CGCD)HD</u>	$\omega_1(^{13}\text{C}/^1\text{H})$	6.3; 95	5.3	(0.45/0.33)
(¹³ C)				
¹ H-TOCSY- <u>HCH-COSY</u> [†]	$\omega_1(^{13}\text{C}/^1\text{H})$	15; 150	3.4	(0.76/-)

One millimolar solution of "Z-domain" of *Staphylococcal* protein A at T = 25°C. The radio-frequency (rf) carrier for ¹H-frequency labeling in the projected "HC"-dimensions in which the chemical shifts of the aliphatic moieties are measured was set to 0 ppm. In 2D ¹H-TOCSY-HCH-COSY, the ¹H rf carrier was set to the position of the water line throughout. t_{\max} denotes the maximal evolution time.

The suite of experiments in this table can provide complete resonance assignments of proteins, excluding only the side chain NH_n moieties, the CH^r groups of histidinyl, and the CH³, CH^{2,3}, and CH^{1,2} groups of tryptophanyl residues (which can be obtained as described in ref. 17). Notably, Z-domain does not contain tryptophans.

* Approach 1: Use of incomplete polarization transfer (rows labeled with "INEPT"); Approach 2: use of ¹³C steady state magnetization (rows labeled with "¹³C").

[†] Direct dimension: $t_{\max} = 73$ ms/512 complex points.

[‡] The average signal-to-noise (S/N) ratio of peaks observed in this subspectrum was 33.2.

[§] The increment for ¹³C^a chemical shift evolution was scaled by a factor of 0.5 relative to the values used to sample ¹³C=O evolution (5).

[¶] The mixing times for the ¹³C-TOCSY relay was set to 21 ms. The S/N ratios for the double-relay central peaks were too low to be accurately evaluated.

[¶] The mixing time for the ¹H-TOCSY relay was set to 25 ms. The acquisition of central peaks is prevented by the use of spin-lock purge pulses (flanking the total correlation relay) to obtain pure phases.

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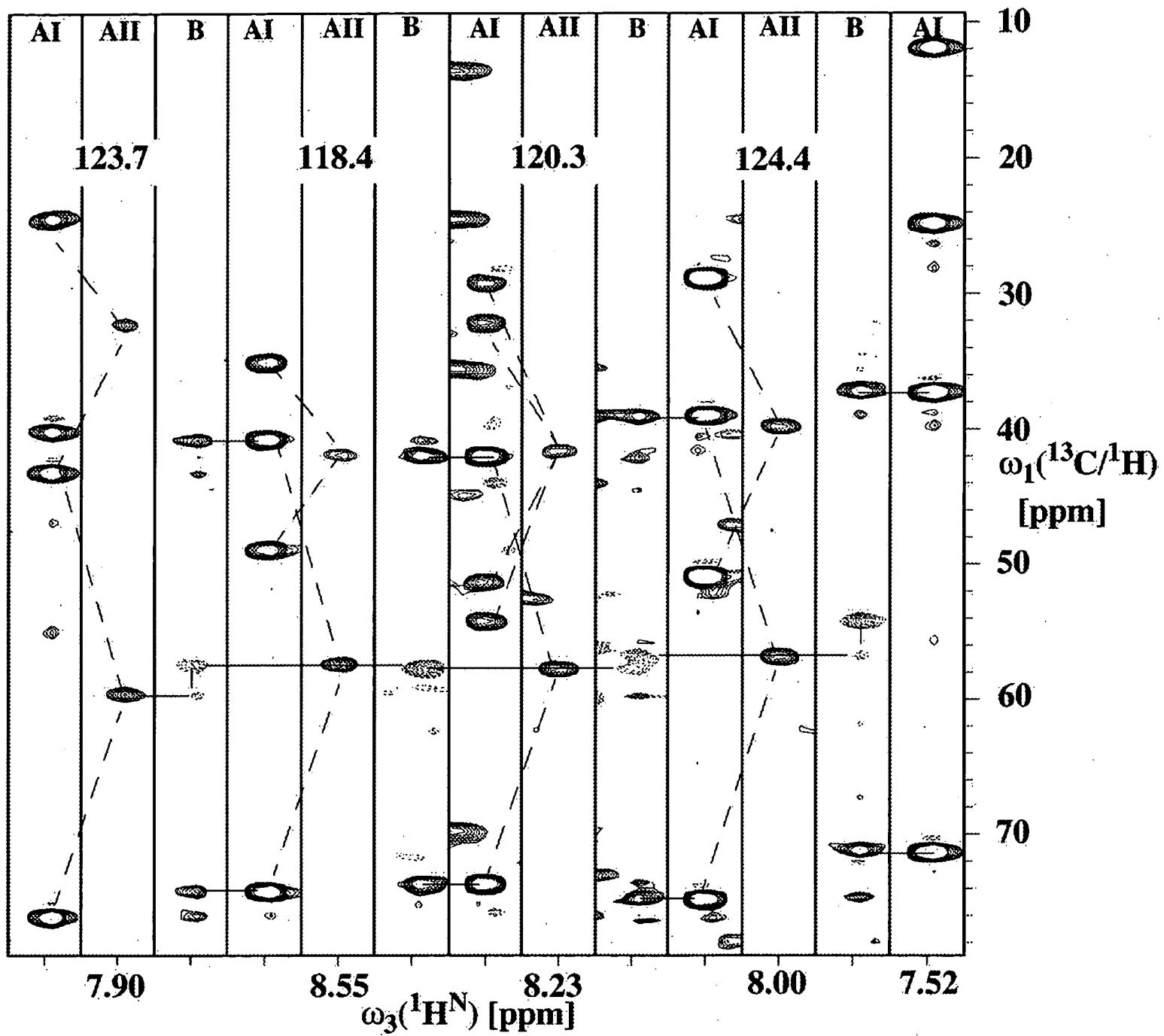
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Asp53

Ala 54

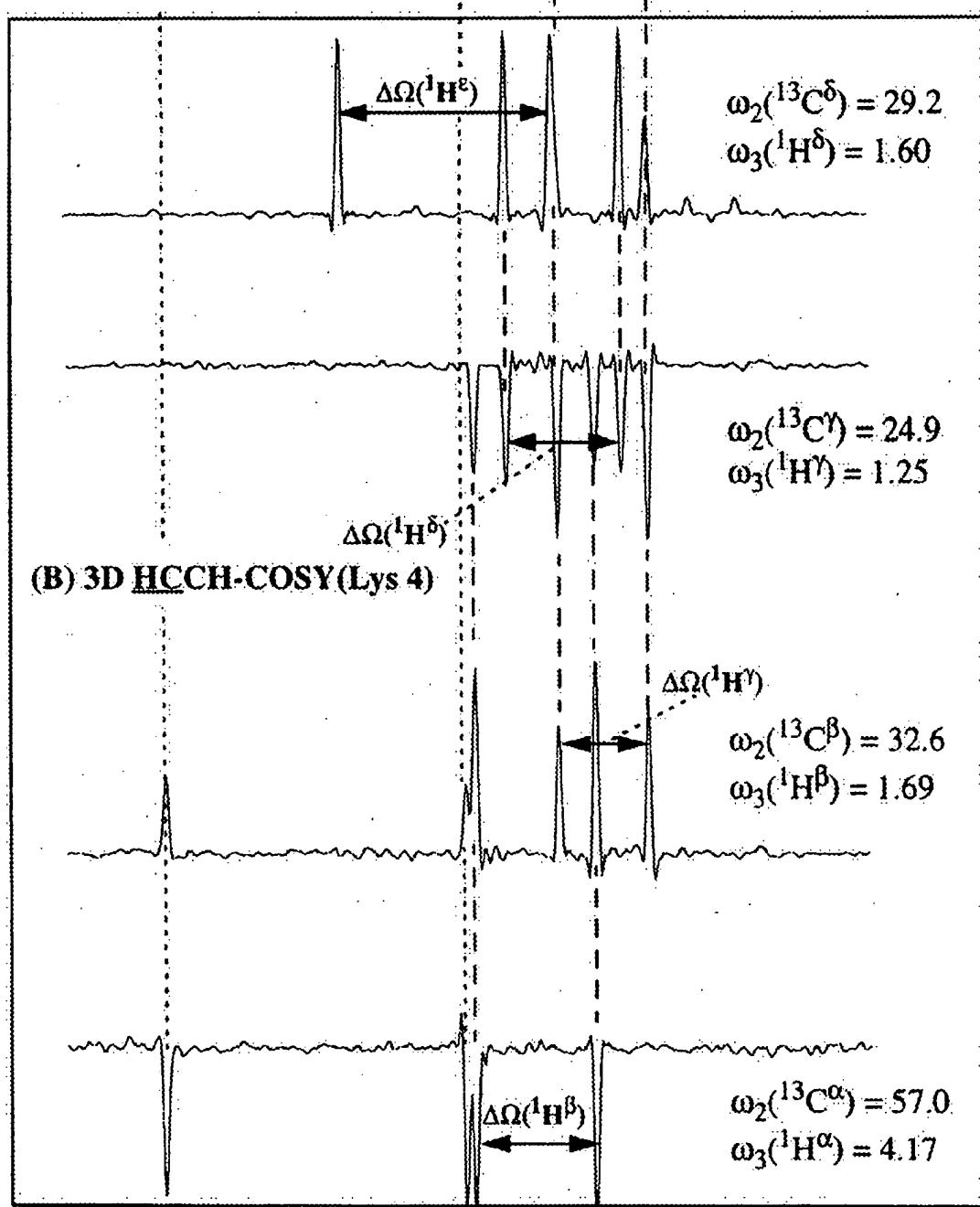
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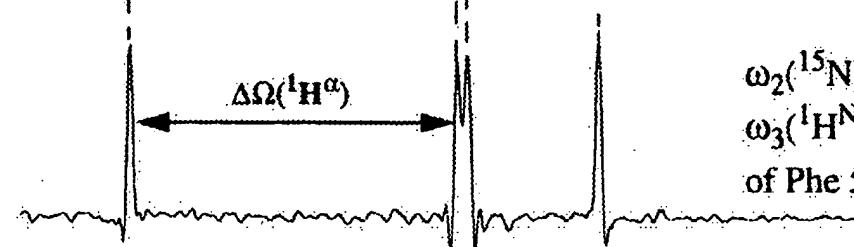


$\omega_2(^{13}\text{C}^{\gamma}) = 24.9$
 $\omega_3(^1\text{H}^{\gamma}) = 1.25$

(C) 3D HCC-TOCSY(Lys 4)

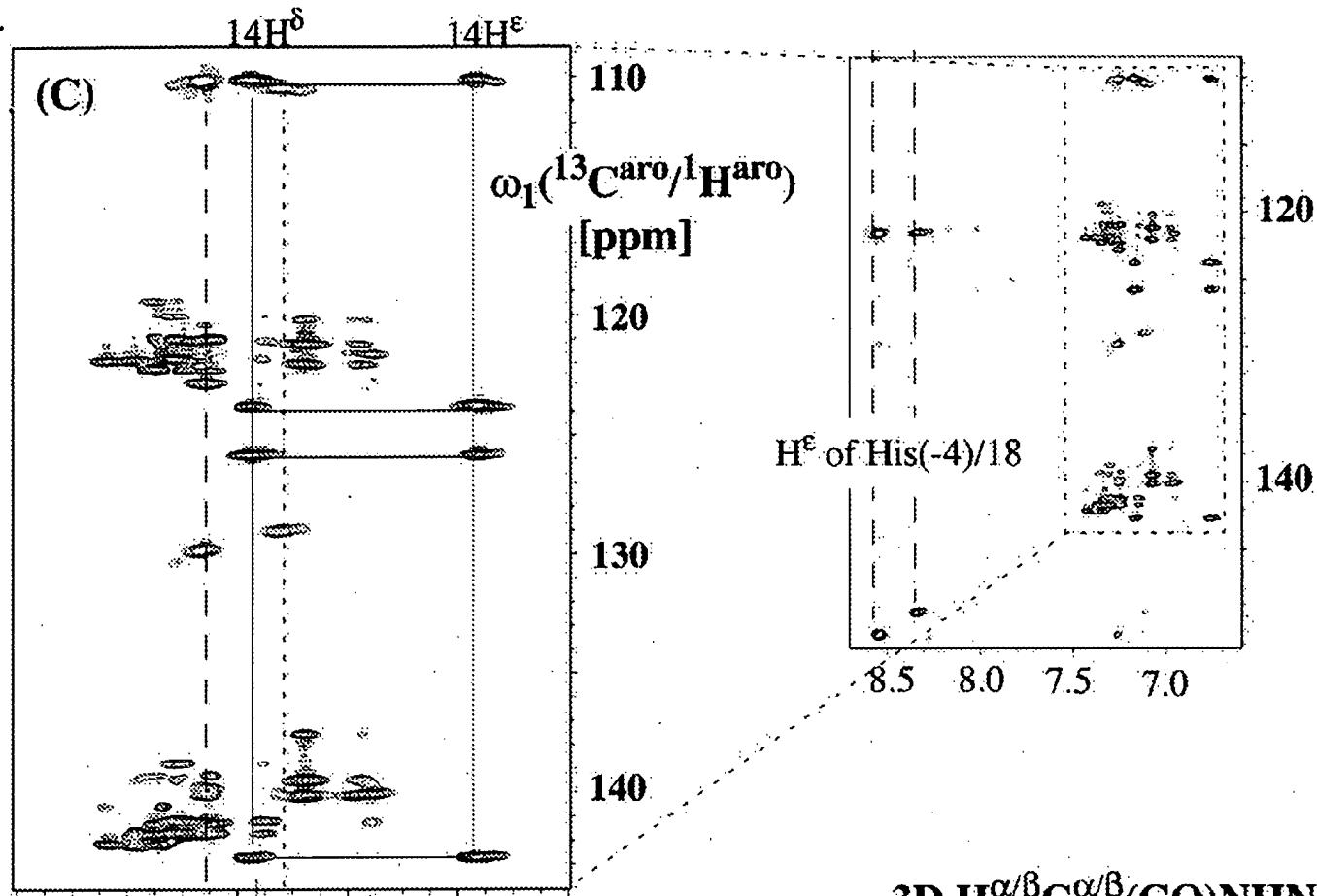


(A) 3D H^{αβ}C^{αβ}(CO)NHN

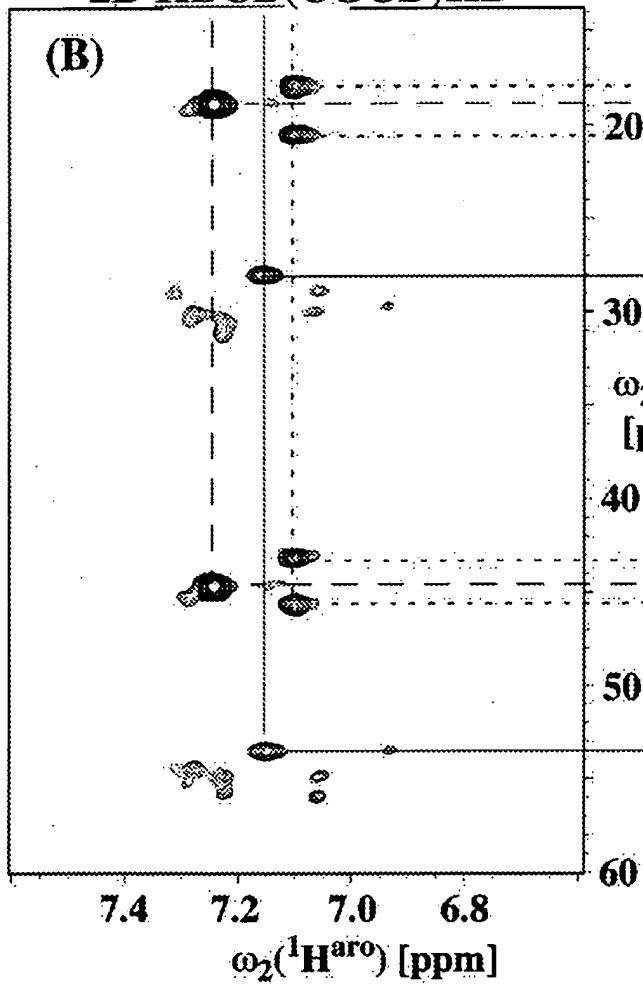


80 70 60 50 40 30 20 10 0
 $\omega_1(^{13}\text{C}/^1\text{H}) [\text{ppm}]$

2D ^1H -TOCSY-relayed HCH-COSY



2D HBCB(CGCD)HD



3D H^α/β - C^α/β (CO)NHN

His(-4) Phe5 Phe13 Tyr14 His18 Phe30

